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PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.:

10/771,692

Filing Date:

February 4, 2004

Applicant:

PARK, Edward H. et al.

Group Art Unit:

3673

Examiner:

Alison K. Pickard

Title:

Dynamic Seal Using Vulcanization of Fluorocarbon

Elastomers

Attorney Docket:

03-0052 (8470-000016)

DECLARATION UNDER 37 C.F.R. § 1.131

PURPOSE OF DECLARATION

- 1. We are the inventors who made application for a patent on February 4, 2004 having the subject matter described and claimed therein, including Claims 1 through 30.
- 2. This declaration is being presented to establish conception and reduction to practice of the invention of our above identified patent application in the United States at a date prior to July 15, 2003, the filing date of U.S. Pat. No. 7,022,769.

FACTS AND DOCUMENTARY EVIDENCE

3. The conception and reduction to practice of our invention in the United States is evidenced by the notebook pages, interim report, and Invention Disclosure Form attached hereto at Exhibit A through Exhibit F. These pages are true copies of the original documents or

electronic files. All copies of notebook pages are from the supporting lab notebook(s) kept by Edward Park in the normal course of business. As indicated below, any redacted portions of the Exhibits either disclose dates that are prior to July 15, 2003, dates that are shortly after July 15, 2003, or disclose personal or confidential client information.

- 4. Prior to July 15, 2003, our invention was conceived and reduced to practice in the United States and experiments were conducted to generate the data detailed in Exhibit A through Exhibit F.
- 5. The notebook pages provided at Exhibit A evidence various formulations of fluorocarbon elastomers used in accordance with the teachings of the present invention. The redacted portions of Exhibit A either disclose dates prior to July 15, 2003 or personal or confidential client information.
- 6. The work order provided at Exhibit B evidences that having earlier conceived of the concept of the present invention, we requested various material development testing to generate data concerning the seals of the present invention. The redacted portions of Exhibit B either disclose dates prior to July 15, 2003 or personal or confidential client information.
- 7. The notebook pages provided at Exhibit C evidence reduction to practice, experimental data, and improvements in the design of the present invention. The reducted portions of Exhibit C either disclose dates prior to July 15, 2003 or personal or confidential client information.
- 8. The notebook pages provided at Exhibit D evidence ongoing experimental data and long-term testing of the present invention. The redacted portions of Exhibit D either disclose dates shortly after July 15, 2003 or personal or confidential client information.

- 9. The Interim Report provided at Exhibit E evidences a summary of the previous testing concerning the present invention. The redacted portions of Exhibit E either disclose dates shortly after July 15, 2003 or personal or confidential client information.
- 10. The conception and reduction to practice of our invention in the United States is further evidenced by the written description attached at Exhibit F. These pages were taken from an Invention Disclosure Form that summarizes the previous findings and data concerning the invention of the pending patent application. The redacted portions of Exhibit F either disclose dates that are prior to July 15, 2003, or are shortly after.

Declaration Under 37 C.F.R. § 1.131

DECLARATION

Each of the undersigned hereby declares that the statements made herein are of his own knowledge and true and that any statement made on information and belief is believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statement may jeopardize the validity of the application, and any patent issuing thereon, on his patent to which this declaration is directed.

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Edward Hosung Park	Date
Alexander Berdichevsky	8/17/06 Date
MM.	8/17/06
Vahidin Alajbegovic	Date

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Work Order Number: 7384-021030
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3013-030421

Seal Test

Project Name:	Seal Test		
Work Order #:	3013-030421	PATTH#	

Lead Center Location Code	PLY - Material 1 6317	Development -	Requestor's Name	David Barth
Requested Start Date			Requestor's Phone Number	734.354.5377
Requested Completion Date			Primary Investigator	Cory Johnson
Promised Completion Date			Chemist of Record	N/A
Work Type	М		Report Copy List	CAM@FNGP.COM
Material Family				
Completion	Status	COMPLETE		Update Completion Status

	Statement of Work										
Test for a	Seal provided per test procedure Cory - Please see Chris Mains dvise and training on setting up the test.										
×	The supplied samples or components are not customer owned.										
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	Disposition of samples:										
	The work will be completed by the Mechanical Lab.										

Hours logged for the project = 155 hrs.

Comments by the Investigator



Date		The second secon
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A · · · · · · · · · · · · · · · · ·	O:02 AM Subject: Shaft Seal Test with New Injection Molding Grade Fluoroprenes	
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rakvedil va 4 + d de Anie 6 militarraksinskan + +	Edward Park —— Forwarded by Edward Park/Plym/North/FNGP on 10:01 AM ——	~
i	Edward Park To: Cory Johnson/Plym/North/FNGP@FNGP, James Bronersky/Plym/North/FNGP@FNGP	
7	10:00 AM CC: Joe Walker/Plym/North/FNGP@FNGP Subject: Shaft Seal Test with New Injection Molding Grade Fluoroprenes	
	Cory and Jim,	12 -
- A	As we discussed this morning, I understand that shart seal made with 008-B series (B, BB and BBB:	4
	As we discussed this monthly in the state that the state of the state	1
	better than shaft seal with three 008-A series materiels (most of them leaked within 2 hours of linet cycle).	1 July
4 . j.	are not sure when the leak was occurred during the overnight run. In the future, we may need some sort of monitoring device, such as time lap video recording device, to detect the moment of leak during the	No
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	Beyond new material development effort, we need a proper shaft seal design, for example, the location of radial spring to provide a right amount of radial force to the proper location of shaft seal, which is mentioned by Dave Sakata. I understand that a design and fabrication of modified shaft seal mod insert is	****
MAY TOO IT IN THE THE BA STREET SAGE	In progress, Cory and Jim, please continue on shaft seal test for two remaining shaft seals with 006-BB and 006-BBB formulations.	** ** ** ** **** *
	Edward Park	
tanti tapan taleh dibe ar-ar-ar-ar-ar-ar-ar-	* Shaft Seal Test *	
· · · · · · · · · · · · · · · · · · ·	Rear Pinion (M80) Test - 4 seals/40 cycles each	
	1. Dynamic CCW 1 min. 180oF 500 rpm 2. Dynamic CW 2 hrs. 225oF 2000 rpm 3. Dynamic CW 16 hrs. 225oF 4600 rpm	
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÷: !* PROJECT NAME FRM-TPU RSS. REDACTED pre heat-treatment before tachal Shaff seal test implemented to extend the 17 fe of RSS, which faces up to 275° / Juing the lest cycle the plastic nature of Fla-TPV is caused for shrinkage and expansion of shaff stal material during the test cycle. Pre heat treatment process was applied to refease the residual stress during the injection molding process. The guich solidification of multen plantics in the cold mold at the high pressure generate tozen residual Stress during the insection molding process The release of frozen Lesidial Stress before the RSS tect is necessary to mminize the Imensional fluctuation during the elevated temperature cycle. 5KM-TPV bated 255 are pre heattreated of 12t°C and 150°C SIGNATURE . READ AND UNDERSTOOD

	REDACTED CONTRACTOR
227 PROJECT NA	ME RKM-TPU RSS. NOTEBOOK NO.
F.KM-	TPV based RSSs were testeal
I after	heat-treatment. Heat-treatment
is ke	equired to stabilite the dimensional
Fluct	uation due to shrink ge of them-Th
Edwa	rd Park To: Joe Walker/Phym/North/FNGP@FNGP
	10:41 AM Subject: FKM-TPV Radial Shaft Seal (RSS) Test
protocol attached molding grades of	you know that our RSS test is currently 3rd cycle without a sign of any leaks. The test it below for your information. As I mentioned, these RSSs were molded with new injection of FKM-TPV formulations and heat-treated (annealed) at 300oF (150oC) to eliminate the repeated high and low temperature cycle. It looks like this approach is working well at will mold more RSSs and heat-treat them to confirm the repeatability.
We also received	d five extrusion grades of FKM-TPVs (about 50 lbs each) from outside contract We will confirm the extrudability of these internal tube extrusion trial and send a couple of best performing grades to extrusion trial.
that temperature ~ 2000F higher to molding operation our current model.	hirdiogy, which could help to achieve the now osaance for multi-cavity field. They cannot be build-up at the runner surface with the high speed injection operation could reached 100 than the center of the runner. This behavior maybe closely related for our rubber injection on, which we are trying to confirm high temperature generation at the high shear rate with logical study. By the way, I completed the shear viscosity measurement as a function of three rubbers (FKM, ACM and Silicone). I am still waiting for new die set to measure the as a function of high shear rate (up to 7,000 1/S).
FKM-TPV we st time, since it red for this project,	to discuss the adhesive development for our FKM-TPV had two new adhesive formulations, and they molded adhesive coated coupons with upplied in front of us. We could not find out the effectiveness of these adhesives at that quiried 24 hours waiting period before testing. We met several key chemists and managers and we decided to have more frequent contacts each other to inform new adhesive and iopment, respectively.
Edward Park	
* Shaft Seal Te	st °
1, Dynamic CC	W 1 min. 180oF 500 rpm
2. Dynamic CV 3. Dynamic CV 4. Dynamic CV	V 16 hrs. 225oF 4600 rpm V 1hr. 59 min. 275oF 3000 rpm
5. Static	4 hrs. No Heat
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	performance of Fluc conventional elasto	mer RSS and Fluor	roprene RS	S, such as	thermoplast	ic nature of	Fluoroprenontact area	e, material and shape		
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-613	due to mold flow de seal, etc. I think you design to accomed	sign during injectors to could get some h	elp from nev aterial. The	w seal eng	neer, Al Ala asurements	begovic, to of Fluorop	modifiy cur rene RSS b	rent RSS efore and	•	1
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	Sample Material	Post Cure		test Condi	ions w/spring	Post	-test Condit Load	ions w/spring	Time Failed	
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	2 PCC550-150A 3 PCC550-150B	125 C 150 C	62.6 62.7	40.0 15.0	65.0 23.0	63.4 63.4	2.0	4.0 4.5	2.3hrs 69.0hrs	
	4 PCC550-150A 5 PCC550-150B	150 C 150 C w/center		65.0	65.0 85.0	63.4 63.3	3.0	8.1 16.0	2.2hrs 19.5hrs	
	6 PCC550-150B 7 PCC550-150A	150 C w/center 150 C w/center	N/A	85.0 50.0	55.0	63.4 63.6	1.6	2.2	43.1hrs 43.1hrs	1
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PROJECT NAME TPE based Dynamic NOTEBOOK NO. 0:37AM nd Seal Jesipus are proposed TPE type material. Intrally tikm-TPU type TIE materials are ma) or interest to Levelop Lynamic seds to accommodate the properties of Flore - TPU (Fluoropiene) type materials. Hybrid seal designs are the combination of typical commencial seal dosign for elastomer (nubber) and pTET plastic materials, since the properties of 7PE type materials are the mixture of elastomer and PTEE plastic material properties. The proposal Seal designs are as follows; Structure Structure SIGNATURE . DATE READ AND UNDERSTOOD

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Freudenberg-NOK
Plymouth Technology Center
Dynamic sealing R&D
47690 E. Anchor Court
Plymouth, MI 48170



Fluoroprene™ Radial Shaft Seal Test
-Interim Report-

In order to better understand FluoropreneTM material in a dynamic sealing application, seal design used was to explore the potential use of the new material in some of the current sealing applications. RSS originally uses FKM (VG705) as the sealing material. FluoropreneTM was used as a direct replacement. 38 seals were injection molded in the existing test mold. Three FluoropreneTM formulations were used: PCC550-150A (basic), PCC550-150AA (addition of wear package) and PCC550-150AAA (with no carbon black). In absence of an adequate adhesive, 64 holes were drilled in the metal case to allow for mechanical bond between the elastomer and the metal case. Heat treatment was performed on the seals to allow for stress relaxation. Previous experiments showed strong influence of thermal cycling on the elastomer shape and sealing capability. 22 seals were heat treated before trimming the lip and 16 seals were trimmed first and then heat treated. Heat treatment consisted of keeping the seals for 16 hours in an oven at 150°C. Additional heat treatment of 4 hours at 150°C was performed on the seals that were originally first heat treated and then trimmed.

The seals which were heat treated first exhibited the deformation as shown in Figure 1. Material shrinkage in the center cap pulled the seal lip inward. This presented a problem for trimming operation due to change in geometry of the lip. Approach angle of the trimming knife was modified to produce correctly trimmed lip. This deformation of the lip also affects the R-value (distance between the lip contact and the center of the spring). Too much deformation can create a negative R-value, which is not acceptable.

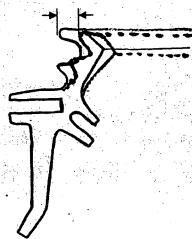


Figure 1: Sketch of Lip Deformation after Initial Heat Treatment

An additional deformation was observed on the dust lip. It is suspected that small differences in the material density and flow pattern in the mold become visible after heat treatment, thus forming an uneven lip as shown in Figure 2.

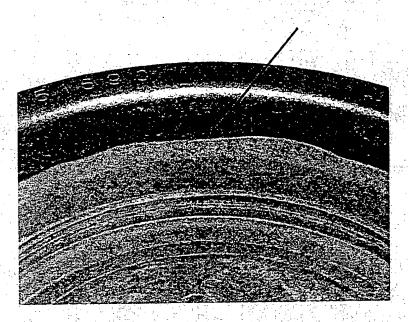


Figure 2: Dust Lip Deformation after Heat Treatment

Final seal lip internal diameters were measured on the OGP. This data is shown in Figure 3. Red color is used for seals molded from PCC550-150A (basic formulation), Blue for PCC550-150AA (addition of wear package) and magenta for PCC550-150AAA (with no carbon black). Squares represent the seals that were first trimmed and then heat treated. Circles represent the seals that were heat treated first, then trimmed and heat treated again. Second heat treatment was necessary to ensure lip achieve stable final dimension.

Several rational subgroups can be derived from the plot.

First group are the seals shown in squares (trim — heat treatment). These seals from all three materials show good dimensional correlation. Mean ID is 64.81 and standard deviation is 0.133.

Second group are the seals shown in red circles (material 150A, heat treated-trimmed-heat treated). The ID is scattered over a much larger range (1.54mm) partly due to difficulty to trim the deformed lip accurately and partly due to compounded error accumulated in the multi staged process (two heat treatments). Mean value for this group is 63.57mm and standard deviation is 0.481.

Third group are the seals shown in blue circles (material 150AA, treated-trimmed-heat treated). This group of seals has low scatter even it had same treatment as the second group. Apparently, material PCC550-150AA (with addition of wear package) exhibits very good dimensional stability. The mean is 63.68 and standard deviation is 0.155.

Fourth group of seals (shown in magenta circles) exhibits similar behavior as seals from the second group. The mean value is 63.78mm and standard deviation is 0.477.

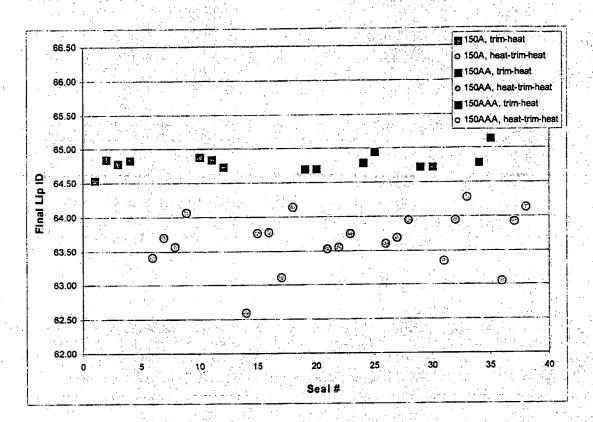


Figure 3: Final Lip ID (mm)

Two Omega test shafts are currently being made for testing of these seals. The shaft dimensions are 65.91 mm and 64.76 mm. The same leakage test cycle will be used for these seals as it was on the previous trials.



FREUDENBERG-NOK INVENTION DISCLOSURE FORM

Su	bmitted By:_Edward Park_	Date:
1.	Short Title of the Invention:_TPE Based Commercial Value, \$_5,000,000	Dynamic Seal Development_& Est.
	Who first thought of the invention: Edw	
	When:	Where:_FNGP
	(attach copy)	Where: FNGP
4.	reports): FNGP Note 1	e) verify the conception date (letters, notes, Book #PCC550 (page 1); FNGP Note Book
	#PCC550 (pages 190 – 191);	FNGP Note Book #PCC550 (page 227); C550 (page 234); FNGF
•	Note Book #PCC550 (page 243)	11 NG1
5.	The invention was first disclosed to a per	rson within FNGP:
	Date:	То:
6.	First disclosure to a person outside of FN	IGP:
	Date:	To:
7.		mposition, or process of the invention (attach
	a. Sample(s) to:	Date:
	b. Sale to:	Date:
8.	Brief description of your invention (attac	h additional sheets if necessary)

Modification of critical material properties required for dynamic seal performance and seal designs to accommodate unique characteristics of TPE materials are proposed to develop TPE based dynamic seals. Typically, two distinctive materials, cured elastomers and PTFE plastics, are widely used for dynamic seal applications. Material properties and commercial seal designs (Figures 1 and 2) for these two materials are quite different for dynamic seal applications. Since the properties of TPE materials are the mixture of elastomers and plastics properties, seal designs could be the combination of elastomers seal design and plastic (PTFE) seal design (Hybrid Seal Design). The initial choice of TPE material is FKM-TPV type TPE, which consists of a two-phase particulate morphology where FKM elastomer phase exists as discrete vulcanized particulates within a continuous FKM thermoplastic fluoroplastic matrix. To determine the required properties and the adequate design for FKM-TPV material, the differences in properties among elastomers, PTFE plastics and FKM-TPV materials should be considered. The examples of differences in properties and seal designs to be considered for FKM-TPV based dynamic seals are as follows, the ratio of recovery time to real time, the ratio of loss modulus to storage modulus, compression set, thermoplastic elastomeric nature of FKM-TPV, wettability with the lubricant in creation of the hydrodynamic film, material

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shrinkage, dimensional stability, radial spring force, lip location against the position of radial spring load, micro pumping effect, contact area and shape (leg length and contour) of lip, surface texture of lip (number of asperities), molecular orientation of FKM-TPV material due to mold flow design during injection molding operation, addition of inert fillers, application of various helix structure in the seal, etc. The wide spectrum of FKM-TPV materials can be obtained by varying the kind and ratio of FKM elastomers and fluoro-plastics, which make it possible to modify material properties to accommodate the effective dynamic seal designs. The desirable properties of TPE materials are as follows, the ratio of recovery time to real time (less than 1), the ratio of loss modulus to storage modulus (less than 0.1), low compression set at the elevated temperature, visco-elastic properties lean to elastic characteristic, high surface wetting capability with the lubricant in creation of the hydrodynamic film (low surface energy), pre heat-treatment to eliminate residual stress during thermoplastic processing, dimensional stability by adding fillers, and low shrinkage material. The seal design parameters should be modified to accommodate the unique properties of TPE materials are as follows, high radial spring force (followability improvement), lip location against the position of radial spring load (promote micro pumping effect), large contact area and shape (long leg length and contour) of lip, surface texture of lip (large number of asperities), molecular orientation of FKM-TPV material due to mold flow design during injection molding operation, addition of inert fillers (improve wear resistance after initial wear) and application of various helix structure in the seal (additional leakage protection). Based on the consideration of desirable properties and seal design parameters for TPE type materials, three hybrid seal designs are proposed for general TPE type materials (Figure 3). Initially, these designs are applied to FKM-TPV type TPE materials.

9. What advantages does your idea have (attach additional sheets if necessary)

The modification of critical material properties by developing a variety of formulations with the combinations of various FKM elastomers and fluoro-plastics and the modification of seal designs to accommodate the unique characteristics of TPE type materials provide the opportunity to develop TPE based dynamic seals. FKM-TPV type TPE materials under development at FNGP are the initial targets for this concept.

Signatures of Inventor(s)	Witness Signature(s): The above was rea and understood by	
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Date	Date	

Alex Berdichevsky		
		
	Date	Date

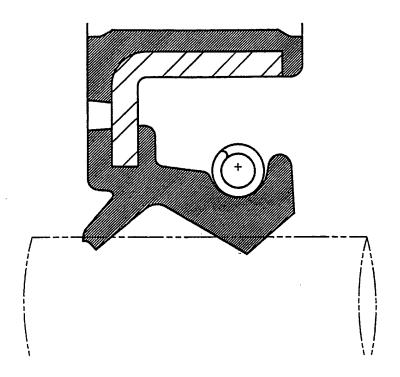


Figure 1. Typical Commercial Rubber (Elastomer) Dynamic Seal

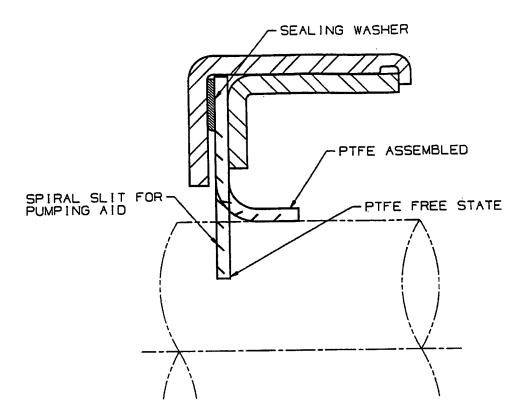


Figure 2. Typical Commercial PTFE Dynamic Seal

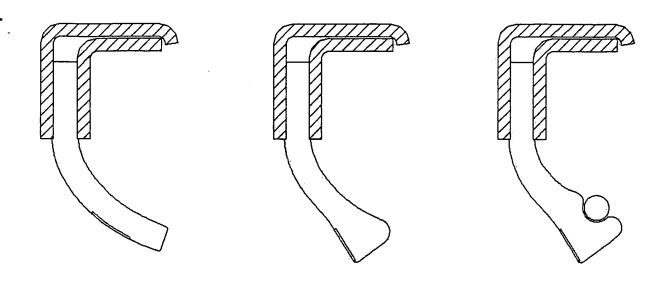


Figure 3. Proposed Hybrid Seal Designs for TPE (FKM-TPV) type Materials

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